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IS 6678 (1972): Ammonium Thiosulphate Solution,
Photographic Grade [CHD 5: Electroplating Chemicals and
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“Knowledge is such a treasure which cannot be stolen”



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IS : 6678 - 1972

Indian Standard
**SPECIFICATION FOR
AMMONIUM THIOSULPHATE SOLUTION,
PHOTOGRAPHIC GRADE**

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

January 1973

Indian Standard
SPECIFICATION FOR
AMMONIUM THIOSULPHATE SOLUTION
PHOTOGRAPHIC GRADE

Photographic Chemicals and Related Materials Sectional
Committee, CDC 44

AMENDMENT NO. 1 MARCH 1989
TO

**IS : 6678 - 1972 SPECIFICATION FOR AMMONIUM
THIOSULPHATE SOLUTION, PHOTOGRAPHIC
GRADE**

[*Page 4, Table 1, Sl No. (iii), col 3*] - Substitute '0.3 to 0.8' for '0.3 to 1.5'.

(*Page 5, clause A-2.1.1, heading*) — Substitute '*0.1 N Iodine Solution*' for '*Standard Iodine Solution*'.

(*Page 6, clause A-2.2, line 2*) — Substitute 'conical flask' for 'beaker'.

(*Page 7, clause A-5.1*) — Insert the following new clause after A-5.1:

A-5.1.1 Relative density may also be determined by using a hydrometer of range 1 200-1 400. However, in case of dispute, the density bottle method shall be the reference method.'

(CDC 44)

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Indian Standard
SPECIFICATION FOR
AMMONIUM THIOSULPHATE SOLUTION,
PHOTOGRAPHIC GRADE

Photographic Chemicals and Related Materials Sectional
Committee, CDC 44

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(Continued from page 1)

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Indian Standard
**SPECIFICATION FOR
AMMONIUM THIOSULPHATE SOLUTION,
PHOTOGRAPHIC GRADE**

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 3 October 1972, after the draft finalized by the Photographic Chemicals and Related Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 Ammonium thiosulphate is also known as ammonium hyposulphite. A sixty percent solution in water is used as the rapid photographic fixing agent.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for ammonium thiosulphate solution used for processing of sensitized photographic materials.

2. REQUIREMENTS

2.1 Description — Ammonium thiosulphate solution, photographic grade, shall be a clear, colourless, aqueous solution of ammonium thiosulphate $[(\text{NH}_4)_2\text{S}_2\text{O}_3]$, free from sediment at ambient temperature.

2.2 Ammonium thiosulphate solution shall comply with the requirements prescribed in Table 1 when tested according to the methods given in Appendix A.

*Rules for rounding off numerical values (*revised*).

**TABLE 1 REQUIREMENTS FOR AMMONIUM THIOSULPHATE SOLUTION,
PHOTOGRAPHIC GRADE**
(Clause 2.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Assay [as (NH ₄) ₂ S ₂ O ₃], percent by mass	59.0 to 61.0	A-2
ii)	Sulphite [as (NH ₄) ₂ SO ₃], percent by mass, Max	1.0	A-3
iii)	Alkalinity (as NH ₄ OH), percent by mass	0.3 to 1.5	A-4
iv)	Relative density at 20/20°C	1.32 to 1.34	A-5
v)	Sulphide [as (NH ₄) ₂ S], percent by mass, Max	0.000 5	A-6
vi)	Insoluble matter and matter precipitated as oxalates, phosphates and hydroxides, percent by mass, Max	0.2	A-7
vii)	Residue on ignition, percent by mass, Max	0.2	A-8
viii)	Iron (as Fe), percent by mass, Max	0.001	A-9
ix)	Heavy metals (as Pb), percent by mass, Max	0.001	A-10

3. PACKING AND MARKING

3.1 Packing— The material shall be packed in glass or plastic (polyethylene) carboys or in such other containers as agreed to between the purchaser and the supplier.

3.2 Marking

3.2.1 The containers shall be securely closed and marked with the manufacturer's name and trade-mark, if any; and the year of manufacture.

3.2.2 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

A P P E N D I X A

(Clause 2.2, and Table 1)

METHODS OF TEST FOR AMMONIUM THIOSULPHATE SOLUTION, PHOTOGRAPHIC GRADE

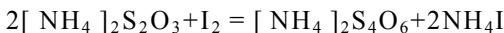
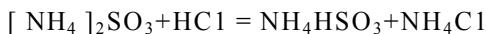
A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise pure chemicals and distilled water (*see IS : 1070-1960**) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF AMMONIUM THIOSULPHATE

A-2.0 General — The determination of thiosulphate content is based on the amount of iodine consumed by the sample less than that consumed by ammonium sulphite present as an impurity. The amount of the latter is determined indirectly by the titration of the ammonium bisulphite that is formed in the course of reaction. The mechanism of the reactions is as follows:



A-2.1 Reagents

A-2.1.1 Standard Iodine Solution — Transfer about 13 g of iodine into a solution of 36 g of potassium iodide in 100 ml of water. After solution is complete, dilute with water to one litre in a volumetric flask.

A-2.1.2 Starch Indicator — Stir 5g of soluble starch with 100 ml of 1 percent salicylic acid solution. Add 300 to 400 ml of boiling water and boil until the starch dissolves. Dilute to one litre with water.

*Specification for water, distilled quality (*revised*).

A-2.1.3 Standardization of Iodine Solution — Place about 0.2 g of powdered arsenic trioxide (As_2O_3), which has been dried overnight over sulphuric acid in a desiccator on a tared 25 mm diameter watch glass and weigh accurately. Transfer the watch glass and contents to a 500-ml glass-stoppered conical flask and dissolve the arsenic trioxide in a mixture of 25 ml of water and 5 ml of 10 percent sodium hydroxide solution. If necessary, warm on a steam bath to complete solution. Cool, acidify slightly with 6 to 7 ml of 10 percent sulphuric acid and add 40 ml of saturated sodium bicarbonate solution. Titrate with the standard iodine solution adding 5 ml of starch indicator just before the end point is reached.

A-2.2 Procedure — Accurately weigh about 8 g of sample into a 250-ml beaker and dilute it to about 100 ml with water. Add a few drops of methyl red indicator and titrate the solution immediately to the first colour change with 0.1 N hydrochloric acid. Dilute to 500 ml with water in a graduated flask and mix well. Take a 50 ml aliquot, and 75 ml of water and 2 ml of starch indicator and titrate the mixture with the standard iodine solution.

A-2.2.1 Add, dropwise, sufficient quantity of the sample solution just to destroy the blue colour, followed by a few drops of methyl red indicator and titrate the mixture with 0.1 N sodium hydroxide solution to the first colour change.

A-2.3 Calculation

$$\text{Ammonium thiosulphate, percent by mass} = \frac{14.82 [V_1 - 0.667 V_2]}{M}$$

where

V_1 = volume in millilitres of 0.1 N iodine solution used,

V = volume in millilitres of 0.1 N sodium hydroxide used, and

M = mass in grams of the sample taken for the test.

A-3. DETERMINATION OF SULPHITE

A-3.1 Calculation

$$\text{Sulphite [as } (\text{NH}_4)_2 \text{SO}_3 \text{], percent by mass} = \frac{3.88 V_2}{M},$$

where

V_2 = volume in millilitres of 0.1 N sodium hydroxide used in **A-2.2.1**, and

M = mass in grams of the sample taken for the test in **A-2.2**.

A-4. DETERMINATION OF ALKALINITY

A-4.1 Calculation

$$\text{Alkalinity (as NH}_4\text{OH), percent by mass} = \frac{0.35 \times V}{M}$$

where

V = volume in millilitres of 0.1 N hydrochloric acid required in A-2.2.1, and

M = mass in grams of the sample taken for the test in A-2.2.

A-5. DETERMINATION OF RELATIVE DENSITY

A-5.1 Relative density shall be determined by density bottle method in accordance with IS : 4730-1968*.

NOTE — This test is not to be used as method of assay, but is intended as a means of ensuring the absence of significant quantities of inert materials and other thiosulphates.

A-6. DETERMINATION OF SULPHIDE

A-6.1 Apparatus

A-6.1.1 Nessler Cylinders — 50 ml capacity.

A-6.2 Reagents

A-6.2.1 Alkaline Lead Solution — Dissolve 10 g lead acetate [$\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$] in sufficient water to produce 100 ml. Add sufficient 10 percent (*m/v*) sodium hydroxide solution to dissolve the precipitate first formed, and then add 5 ml in excess.

A-6.2.2 Standard Sulphide Solution — Dissolve sufficient sodium sulphide crystals in distilled water to give 200 ml containing 1.5 ± 0.01 g sodium sulphide crystals ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$). Dilute one ml of this solution to 100 ml with freshly boiled and cooled distilled water. One millilitre of the resulting solution = 0.021 mg (NH_4S). Do not store this solution but prepare as required.

A-6.3 Procedure — Dilute 20 ± 0.1 g of the sample to 40 ml with water in a 50-ml Nessler cylinder, add 2 ml of the alkaline lead solution, dilute to 50 ml, and mix well. At the same time, take 4.5 ml of the standard sulphide solution, in a similar Nessler cylinder, and dilute to 50 ml with water.

A-6.3.1 The colour produced in the sample solution shall not be stronger than that produced in the standard.

*Method for the determination of density of liquids.

A-7. DETERMINATION OF INSOLUBLE MATTER AND MATTER PRECIPITATED AS OXALATES, PHOSPHATES AND HYDROXIDES

A-7.1 Reagents

A-7.1.1 *Ammonium Oxalate Solution* — 4 percent (*m/v*).

A-7.1.2 *Ammonium Phosphate Solution* — 10 percent (*m/v*).

A-7.1.3 *Ammonium Hydroxide* — 10 percent (*v/v*).

A-7.1.4 *Ammonium Hydroxide* — (1/39) solution.

A-7.2 Procedure — Dilute 20 ± 0.1 g of the sample with 75 ml of water and mix well. Add 10 ml of ammonium oxalate solution, 4 ml of ammonium phosphate solution, and 20 ml of ammonium hydroxide. Allow to stand overnight. If any precipitate is formed, filter and wash with ammonium hydroxide (1 : 39). Dry and ignite at 600 ± 25°C for 4 hours. Cool in a desiccator and weigh.

A-7.3 Calculation

Insoluble matter and matter precipitated
as oxalates, phosphates and hydro-
xides, percent by mass = $\frac{m \times 100}{M}$

where

m = mass in grams of residue, and

M = mass in grams of material taken for the test.

A-8. DETERMINATION OF RESIDUE AFTER IGNITION

A-8.1 Procedure — Evaporate 10 ± 0.1 g of sample to dryness in a weighed crucible and finally ignite at 600 ± 5°C for 4 hours. Cool in a desiccator and weigh.

A-8.2 Calculation

Residue on ignition, percent by mass = $\frac{100 \times M_1}{M}$

where

*M*₁ = mass in grams of the residue, and

M = mass in grams of the sample taken for the test.

A-9. DETERMINATION OF IRON

A-9.1 Apparatus

A-9.1.1 Nessler Cylinders — 50 ml capacity.

A-9.2 Reagents

A-9.2.1 Ammonium Hydroxide — 10 percent (*v/v*).

A-9.2.2 Citric Acid Solution — 20 percent (*m/v*).

A-9.2.3 Hydrochloric Acid — 10 percent (*v/v*).

A-9.2.4 Thioglycollic Acid — containing not less than 89 percent (*m/v*) of thioglycollic acid (C₂H₄O₂S).

A-9.2.5 Standard Iron Solution — Dissolve 7.02 g of crystalline ammonium ferrous sulphate in about 300 ml of water containing 2 ml of sulphuric acid, relative density 1.84, and dilute to 1 000 ml. Further dilute 25 ml to 1 000 ml with water freshly as required. One millilitre of the resulting solution contains 2.5 µg of iron (as Fe).

A-9.3 Procedure — Dissolve the residue obtained in A-8.1 in 10 ml of dilute hydrochloric acid and heat to boiling. Cool and make up to 50 ml with water. Dilute 4 ml of standard iron solution also to 50 ml.

A-9.3.1 Transfer a 10 ml aliquot of the test solution and the iron standard solution to Nessler cylinders and dilute to about 30 ml with water. To both cylinders, add 2 ml of citric acid solution and 2 drops of thioglycollic acid. Mix, make just alkaline with ammonium hydroxide and dilute to 50 ml with water,

A-9.3.2 The limit prescribed shall be taken as not having been exceeded if the intensity of the colour produced in the sample solution is not greater than that produced in the standard.

A-10. DETERMINATION OF HEAVY METALS

A-10.1 Apparatus

A-10.1.1 Nessler Cylinders — 50 ml capacity.

A-10.2 Reagents

A-10.2.1 Ammonium Hydroxide — 10 percent (*v/v*).

A-10.2.2 Hydrochloric Acid — approximately 0.1 N solution.

A-10.2.3 Hydrogen Sulphide — saturated solution, freshly prepared.

A-10.2.4 p-Nitrophenol Indicator — 0.25 percent aqueous solution.

A-10.2.5 Standard Heavy Metals Solution — Dissolve 0.64 g of lead nitrate in water, add 10 ml of nitric acid, relative density 1.42 and dilute to 1 000 ml. Further dilute 25 to 1 000 ml freshly as required. One millilitre of the latter solution contains 10 µg lead (as Pb).

A-10.3 Procedure — Transfer 10 ml aliquot of the test solution prepared as in A-9.3 and 25 ml of the heavy metals standard, each to a Nessler cylinder, add 2 drops of the *p*-nitrophenol indicator, and make slightly alkaline with ammonium hydroxide. Add hydrochloric acid solution drop-wise to each until the colour of the indicator is just discharged, then add 1 ml in excess, followed by 5 ml of the hydrogen sulphide solution. Dilute to 50 ml and mix well.

A-10.3.1 The limit prescribed shall be taken as not having been exceeded if intensity of the colour produced in the sample solution is not greater than that produced in the standard.

APPENDIX B (Clause 4.1)

SAMPLING OF AMMONIUM THIOSULPHATE SOLUTION, PHOTOGRAPHIC GRADE

B-1. SCALE OF SAMPLING

B-1.1 Lot — All the containers in a single consignment of the material drawn from a single batch of packing shall constitute a lot. If the consignment is found to consist of different batches of packing, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-1.2 For ascertaining the conformity of the material to the requirements of this specification, samples shall be tested separately for each lot. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

B-1.2.1 In order to ensure randomness of selection, use of random sampling tables (*see IS : 4905-1968**) is recommended. If the tables are not available, the following procedure may be used:

Arrange all the containers in the lot in a systematic manner and starting from any one, count them as 1, 2, . . . , r where r is the integral part of N/n (N and n being the lot size and sample size, respectively).

*Methods for random sampling.

Every r th container thus counted shall be withdrawn to constitute the sample.

TABLE 2 NUMBER OF CONTAINERS TO BE CHOSEN
(Clause B-1.2)

LOT SIZE	SAMPLE SIZE
N	n
(1)	(2)
Up to 40	5
41 to 110	7
111 „ 300	10
301 „ 800	15
801 and above	20

NOTE—In the case of lots of size less than 5, all the containers of the lot shall be included in the sample.

B-2. PREPARATION OF TEST SAMPLES

B-2.1 From each of the containers selected according to **B-1.2.1**, a representative portion of the material, sufficient for carrying out the tests specified under **A-3**, shall be drawn separately.

B-2.2 From each of the portions of the material drawn according to **B-2.1**, a small but equal quantity shall be taken and thoroughly mixed to form a composite test sample.

B-2.3 The remaining portion of the material drawn from each container shall be transferred to separate bottles and labelled with full identification particulars on the bottles. The material in each individual bottle shall constitute an individual test sample.

B-3. NUMBER OF TESTS

B-3.1 Tests for the determination of assay (*see 2.2*) of the material shall be conducted on each of the individual test samples and the test results shall be recorded separately for different test samples.

B-3.2 Tests for the requirements of all other characteristics given in Table 1 shall be carried out on the composite test sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 From the test results recorded according to **B-3.1**, the mean (\bar{x}) and range (R) of test results for any characteristic shall be computed as

follows:

$$\text{Mean } (\bar{x}) = \frac{\text{Sum of test results } 1, 2, \dots, n}{\text{Number of test results, } n}$$

Range (R) = Difference between the maximum and minimum of the test results.

B-4.2 In the case of samples of size 10 or more, every 5 test results shall be grouped together and the range of each group shall be determined. Average range (\bar{R}) shall then be computed from the group ranges as given below:

$$\text{Average range } (\bar{R}) = \frac{\text{Sum of group ranges}}{\text{Number of groups}}$$

B-4.3 The lot shall be declared as conforming to the requirements of the specification for different characteristics if the corresponding criteria for conformity given below are complied:

<i>Characteristic</i>	<i>Criteria for Conformity</i>
a) Assay	$(\bar{x} - 0.6 R^*)$ shall be greater than or equal to the relevant limit prescribed in 2.2.
b) Those listed in Table 1	Test results on the composite test sample shall meet the corresponding requirements given in Table 1.

*When the sample size is 10 or more, average range (\bar{R}) (see B-4.2) shall be used in place of range (R).

INDIAN STANDARDS

ON

PHOTOGRAPHIC CHEMICALS

IS:

- 246-1972 Sodium thiosulphate, crystalline (*third revision*)
247-1972 Sodium sulphite, anhydrous (*third revision*)
248-1971 Sodium bisulphite (sodium metabisulphite) (*second revision*)
332-1967 Chromium potassium sulphate (chrome alum) (*first revision*)
388-1972 Hydroquinone, photographic grade (*second revision*)
500-1972 Potassium metabisulphite, photographic grade (*second revision*)
557-1968 Sodium acetate, technical and photographic (*first revision*)
2211-1972 Anhydrous sodium thiosulphate, photographic grade (*first revision*)
2318-1963 Silver nitrate, photographic grade
2797-1964 Potassium bromide
4173-1967 4-Methylaminophenol sulphate
5379-1969 Ammonium thiosulphate, photographic grade
5380-1969 Sodium bromide, photographic grade
5381-1969 Quantity packaging of sensitized photographic materials
5431-1969 Definition of motion-picture safety films
6139-1971 Sizes of photographic paper for general use
6212-1971 Method for the determination of residual thiosulphate in processed black and white photographic films and plates
6678-1972 Ammonium thiosulphate solution, photographic grade

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